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(54)【発明の名称】 マグネシウム-イットリウム系水素吸蔵合金の製造方法

(57)【要約】

【課題】 組成が均質で、所望の水素吸蔵特性を有するマグネシウム-イットリウム系水素吸蔵合金を効率よく製造する方法を提供すること。

【解決手段】 イットリウム原料又はイットリウム原料とマグネシウム原料の一部とを溶解炉に装入し、イットリウム原料の融点以下の温度で加熱する第一工程、第一工程の加熱原料に所定の組成比になるようにマグネシウム原料を添加し、好ましくは平均昇温速度が3~12℃/分、圧力上昇速度が10~50 Torr/分になるように加熱溶解する第二工程、及び第二工程で形成した合金溶湯を、好ましくは50~500℃/秒の冷却速度で冷却し、凝固させる第三工程を、順次行うことにより、マグネシウム-イットリウム系水素吸蔵合金を製造する。

【特許請求の範囲】

【請求項1】 マグネシウムとイットリウムを主成分とする水素吸蔵合金を製造するに当たり、(a) イットリウム原料を溶解炉に装入し、その融点以下の温度で加熱する第一工程、(b) 第一工程の加熱原料に、所定の組成比になるようにマグネシウム原料を添加して加熱溶解する第二工程、及び(c) 第二工程で形成したマグネシウム-イットリウム系合金溶湯を冷却凝固させる第三工程を順次行うことを特徴とするマグネシウム-イットリウム系水素吸蔵合金の製造方法。

【請求項2】 マグネシウムとイットリウムを主成分とする水素吸蔵合金を製造するに当たり、(a') イットリウム原料とマグネシウム原料の一部とを溶解炉に装入し、該イットリウム原料の融点以下の温度で加熱する第一工程、(b') 第一工程の加熱原料に、所定の組成比になるようにマグネシウム原料の残部を添加して加熱溶解する第二工程、及び(c) 第二工程で形成したマグネシウム-イットリウム系合金溶湯を冷却凝固させる第三工程を順次行うことを特徴とするマグネシウム-イットリウム系水素吸蔵合金の製造方法。

【請求項3】 第二工程において、マグネシウム原料を添加して加熱溶解する際に、溶解炉内において急激な圧力上昇が起こらないように、炉内圧力の上昇速度を制御しながら加熱溶解する請求項1又は2記載の製造方法。

【請求項4】 溶解炉内の圧力上昇速度を、10～50 Torr/分に制御する請求項3記載の製造方法。

【請求項5】 溶解炉内の圧力上昇速度の制御を、炉内昇温速度を制御することにより行う請求項3又は4記載の製造方法。

【請求項6】 溶解炉内の昇温速度を、3～12℃/分に制御する請求項5記載の製造方法。

【請求項7】 第三工程において、マグネシウム-イットリウム系合金溶湯を50～500℃/秒の冷却速度で冷却し、凝固させる請求項1又は2記載の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】 本発明は、マグネシウム-イットリウム系水素吸蔵合金の製造方法に関し、さらに詳しくは、水素貯蔵、ヒートポンプ、電池などに利用可能な、常圧から10atmに平衡圧を有する高温で使用するタイプの均質組成のマグネシウム-イットリウム系水素吸蔵合金を効率よく製造する方法に関するものである。

【0002】

【従来の技術】 近年、金属系新素材の一つとして水素吸蔵合金が注目され、その応用研究が積極的になされている。この水素吸蔵合金は、例えば温度を低くしたり、圧力を加え、水素を吸収して金属水素化合物となると共に発熱し、逆に温度を高くしたり、圧力を下げると、吸収していた水素を再び放出すると同時に熱を奪うとい

う性質を有している。そのため、このような性質を利用して、水素吸蔵合金を水素の貯蔵やエネルギーの貯蔵、あるいは化学エネルギーと熱エネルギーの交換などに利用が可能であり、例えば水素貯蔵装置、排熱利用のヒートポンプ、ケミカルエンジン、ニッケル-水素電池の電極材料などへの利用が試みられている。

【0003】 このような水素吸蔵合金には、用途に応じて各種元素を組み合わせた合金が知られており、代表的な合金としては、ランタン-ニッケル系、チタン-鉄系、ジルコニウム-マンガン系、マグネシウム-ニッケル系などが挙げられる。これらの水素吸蔵合金の中で、マグネシウム系合金は解離圧が1気圧を示す温度が200℃以上であり、高温で水素が合金内に吸蔵されるため、高温の熱源を用いて水素の吸蔵・放出が可能となる。したがって、このマグネシウム系合金は、例えば工場廃熱を利用した冷・暖房システム用として、あるいはエンジンの廃熱を利用した水素自動車用などとして有用である。このようなマグネシウム系水素吸蔵合金としては、従来上記のマグネシウム-ニッケル系合金のほか、マグネシウムとランタン、セリウムなどの希土類元素とを組み合わせた合金などが知られている。

【0004】 一方、イットリウムとマグネシウムとの合金については、例えば特公昭57-45817号公報、化学工学会第28回秋期大会研究発表講演要旨集第2分冊、第345ページ「Mg-Y合金の水素透過特性」で開示又は報告されているにすぎず、ほとんど研究されていないのが実状である。また、この合金の製造方法については、上記公報及び要旨集においてもほとんど記載がなく、均質化のための溶解法や凝固法については知られていない。

【0005】 ところで、イットリウムとマグネシウムは、それらの融点を比較してみると、前者が1526℃であるのに対し、後者は649℃であって、大きな差を有している。したがって、一般的には、溶解炉で溶解して所定の組成に合金化しにくい金属である。例えば、イットリウムとマグネシウムを溶解炉で溶解しようとする、昇温するに伴い、まずマグネシウムが溶解して坩堝に沈降するが、同時にこのマグネシウムは昇華しはじめ、マグネシウム-イットリウム合金を形成する前に、マグネシウムが気散し、未溶解分のイットリウムが坩堝の底に残り、所定の組成を有する合金が形成できないという好ましくない事態を招来する。このような好ましくない事態を回避するために、予めロス分に相当する量のマグネシウムを余分に配合しておくことが考えられるが、この場合、気散する量が溶解条件によって一定ではなく、結果として、得られる合金の水素吸蔵特性にばらつきが生じたり、性能が充分に発揮されないなどの問題が生じる。

【0006】

【発明が解決しようとする課題】 本発明は、このような

状況下で、通常の方法では均質な合金化が困難である融点差の大きなマグネシウムとイットリウムとを、組成に偏りが生じないように均質に合金化し、所望の性能を有するマグネシウム-イットリウム系水素吸蔵合金を効率よく製造する方法を提供することを目的とするものである。

【0007】

【課題を解決するための手段】本発明者らは、前記目的を達成するために鋭意研究を重ねた結果、マグネシウムとイットリウムとを合金化する際に、特定の溶解法を用いることにより、組成の均質な合金が効率よく得られること、さらに、形成した合金溶湯を冷却凝固させる際に、特定の冷却速度を採用することにより、組成が均質で、かつ緻密な結晶構造を有する合金が得られることを見出した。本発明は、かかる知見に基づいて完成したものである。

【0008】すなわち、本発明は、

(1) マグネシウムとイットリウムを主成分とする水素吸蔵合金を製造するに当たり、(a) イットリウム原料を溶解炉に装入し、その融点以下の温度で加熱する第一工程、(b) 第一工程の加熱原料に、所定の組成比になるようにマグネシウム原料を添加して加熱溶解する第二工程、及び(c) 第二工程で形成したマグネシウム-イットリウム系合金溶湯を冷却凝固させる第三工程を順次行うことを特徴とするマグネシウム-イットリウム系水素吸蔵合金の製造方法を提供するものである。また、本発明は、

(2) マグネシウムとイットリウムを主成分とする水素吸蔵合金を製造するに当たり、(a') イットリウム原料とマグネシウム原料の一部とを溶解炉に装入し、該イットリウム原料の融点以下の温度で加熱する第一工程、(b') 第一工程の加熱原料に、所定の組成比になるようにマグネシウム原料の残部を添加して加熱溶解する第二工程、及び(c) 第二工程で形成したマグネシウム-イットリウム系合金溶湯を冷却凝固させる第三工程を順次行うことを特徴とするマグネシウム-イットリウム系水素吸蔵合金の製造方法をも提供するものである。

【0009】さらに、本発明を実施するための好ましい態様は、

(3) 第二工程において、マグネシウム原料を添加して加熱溶解する際に、溶解炉内において急激な圧力上昇が起らないように、炉内圧力の上昇速度を制御しながら加熱溶解する上記(1)、(2)記載の製造方法、

(4) 溶解炉内の圧力上昇速度を、10～50 Torr/分に制御する上記(3)記載の製造方法、

(5) 溶解炉内の圧力上昇速度の制御を、炉内昇温速度を制御することにより行う上記(3)、(4)記載の製造方法、

(6) 溶解炉内の昇温速度を、3～12℃/分に制御する上記(5)記載の製造方法、及び

(7) 第三工程において、マグネシウム-イットリウム系合金溶湯を50～500℃/秒の冷却速度で冷却し、凝固させる上記(1)～(6)記載の製造方法、である。

【0010】

【発明の実施の形態】本発明の方法で得られるマグネシウム-イットリウム系水素吸蔵合金は、マグネシウムとイットリウムを主成分とするものであり、マグネシウムとイットリウムのみからなる合金であってもよく、これらに、さらに合金化が可能な適当な金属、例えばカルシウム、バリウム、ストロンチウム、アルミニウム、銅、ニッケル、さらにはランタン、セリウムなどの希土類元素などの中から選ばれた少なくとも一種を含有させたものであってもよい。本発明において、上記マグネシウム-イットリウム系合金の原料の一つとして用いられるマグネシウム原料としては、金属マグネシウムを用いてもよく、合金組成によっては、マグネシウム合金、例えばマグネシウム-カルシウム合金などを用いてもよい。一方、もう一つの原料として用いられるイットリウム原料としては、金属イットリウムを用いてもよく、合金組成によっては、イットリウム合金、例えばイットリウム-希土類元素合金などを用いてもよい。さらに、場合により、マグネシウム原料及びイットリウム原料とともに、上記金属の中から選ばれた適当な金属又は合金を用いることができる。

【0011】また、マグネシウム原料及びイットリウム原料と共に、その他の金属を用いて合金化する場合、その他の金属の中で融点などの性状がイットリウムよりもマグネシウムに近いもの、例えばカルシウムやアルミニウムなどの元素は、マグネシウム原料と同様の取扱い方をするのが好ましく、一方、融点などの性状がマグネシウムよりもイットリウムに近いもの、例えばランタン、ニッケル、銅などの元素は、イットリウム原料と同様の取扱い方をするのが好ましい。

【0012】本発明のマグネシウム-イットリウム系合金の製造方法は、以下に示す第一工程、第二工程及び第三工程の3工程から構成されている。まず、第一工程においては、高周波誘導溶解炉、真空溶解炉などの溶解炉内の坩堝に、イットリウム原料の全部、又はイットリウム原料の全部とマグネシウム原料の一部、好ましくは配合量の30%以下の量のマグネシウム原料とを装入し、好ましくは真空排気したのち、アルゴンガスなどの不活性ガスを導入して、炉内の圧力を好ましくは200 Torrを超える程度に維持する。次いで坩堝内に装入した原料を、イットリウム原料の融点以下の温度において、該原料が赤熱するまで加熱し、昇温させる。この際、上記原料の温度が、マグネシウムの融点以上で、650～900℃の範囲になるように加熱するのが有利である。

【0013】次に、第二次工程においては、上記の第一工程で加熱されたイットリウム原料又はイットリウム原

【0020】比較例2

実施例2と同様に、イットリウムとマグネシウムと銅を、合計2000g用い、この全量を高周波誘導溶解炉内の坩堝に装入し、炉内を0.1 Torrまで排気したのち、アルゴンガスを260 Torrまで導入し、加熱を開始した。約13℃/分の昇温速度で85分間加熱し、*

* 溶湯温度が1070℃に達した時点で合金溶湯を水冷定盤上で冷却凝固させた。このようにして得られた合金の上部と下部からサンプリングを行い、ICP発光分析を行い、組成を求めた。結果を第1表に示す。

【0021】

【表1】

第 1 表

		合 金 組 成 (重量%)		
		イットリウム	マグネシウム	銅
目標		(43.24)	(56.76)	(-)
実施例 1	上部	43.07	56.64	-
	下部	43.01	56.74	-
比較例 1	上部	40.65	59.11	-
	下部	38.72	60.99	-
目標		(40.80)	(50.33)	(8.77)
実施例 2	上部	40.83	50.62	8.74
	下部	40.79	50.53	8.76
比較例 2	上部	39.15	51.38	8.75
	下部	37.22	53.37	8.83

【0022】第1表から分かるように、本発明の方法によれば、いずれの元素も、ほぼ目標組成となっており、また、合金の上部と下部とで、組成のばらつきがほとんどない。これに対し、比較例の方法では、目標組成から大きくはずれ、かつ合金の上部と下部とで組成のばらつきがあり、また、坩堝底にイットリウムの未溶解物が残っていた。

【0023】

【発明の効果】本発明の方法によると、マグネシウムがロスしたり、イットリウムの未溶解分が残ることがなく、均質な組成又は均質な組成と緻密な結晶構造を有するマグネシウム-イットリウム系水素吸蔵合金を効率よく

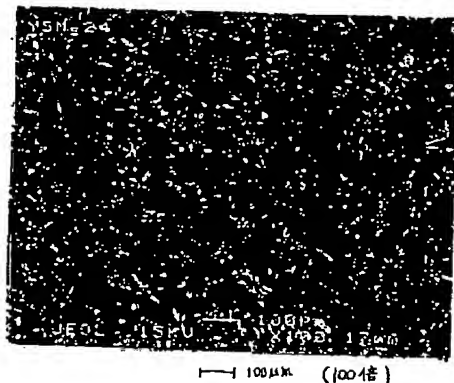
※く製造することができる。本発明の方法で得られたマグネシウム-イットリウム系水素吸蔵合金は、所望の水素吸蔵特性を有しており、例えば工場廃熱を利用した冷・暖房システム用やエンジンの廃熱を利用した水素自動車用などとして有用である。

【図面の簡単な説明】

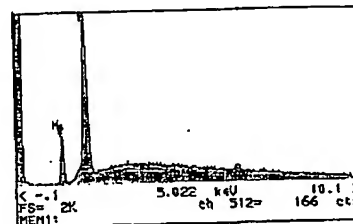
30 【図1】 実施例1で得られたマグネシウム-イットリウム合金の走査型電子顕微鏡 (SEM) 写真図である。

【図2】 実施例1で得られたマグネシウム-イットリウム合金のEDS装置による定性点分析チャートである。

【図1】



【図2】



CLAIMS

[Claim(s)]

[Claim 1] In manufacturing the hydrogen storing metal alloy which uses magnesium and an yttrium as a principal component (a) The first process which inserts an yttrium raw material in a fusion furnace, and is heated at the temperature below the melting point, (b) The second process which adds a magnesium raw material and carries out the heating dissolution so that it may become a predetermined presentation ratio in the heating raw material of the first process, And the manufacture approach of the magnesium-yttrium system hydrogen storing metal alloy characterized by performing the third process which carries out cooling coagulation of the magnesium-yttrium system alloy molten metal formed at the (c) second process one by one.

[Claim 2] In manufacturing the hydrogen storing metal alloy which uses magnesium and an yttrium as a principal component Some of yttrium raw materials and magnesium raw materials are inserted in a fusion furnace. (a') In the heating raw material of the first process and the first (b') process heated at the temperature below the melting point of this yttrium raw material The second process which adds the remainder of a magnesium raw material and carries out the heating dissolution so that it may become a predetermined presentation ratio, And the manufacture approach of the magnesium-yttrium system hydrogen storing metal alloy characterized by performing the third process which carries out cooling coagulation of the magnesium-yttrium system alloy molten metal formed at the (c) second process one by one.

[Claim 3] The manufacture approach according to claim 1 or 2 which carries out the heating dissolution while controlling the climbing speed of furnace internal pressure so that a rapid pressure buildup does not happen into a fusion furnace in case a magnesium raw material is added and the heating dissolution is carried out in the second process.

[Claim 4] The manufacture approach according to claim 3 which controls the pressure climbing speed in a fusion furnace to a part for 10 - 50Torr/.

[Claim 5] The manufacture approach according to claim 3 or 4 of performing control of the pressure climbing speed in a fusion furnace by controlling the programming rate in a furnace.

[Claim 6] The manufacture approach according to claim 5 which controls the

programming rate in a fusion furnace to a part for 3-12-degree-C/.

[Claim 7] The manufacture approach according to claim 1 or 2 of making a magnesium-yttrium system alloy molten metal cooling and solidifying with the cooling rate of 50-500 degrees C/second in the third process.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the approach of manufacturing efficiently the magnesium-yttrium system hydrogen storing metal alloy of a homogeneity presentation of the type used for hydrogen storage, heat pump, a cell, etc. in more detail by the available high temperature which has equilibrium pressure from ordinary pressure at 10atm(s), about the manufacture approach of a magnesium-yttrium system hydrogen storing metal alloy.

[0002]

[Description of the Prior Art] In recent years, a hydrogen storing metal alloy attracts attention as one of the metal system new materials, and the application study is made positively. If it generates heat while it will absorb hydrogen and will serve as metal hydride, if this hydrogen storing metal alloy makes temperature low or a pressure is applied, and temperature is conversely made high or a pressure is lowered, it has the property to take heat at the same time it emits the hydrogen which was being absorbed again. Therefore, using such a property, a hydrogen storing metal alloy can be used for storage of hydrogen, storage of energy, or conversion of chemical energy and heat energy, for example, the use to hydrogen storage equipment, the heat pump of exhaust heat use, a chemical engine, the electrode material of a nickel hydride battery, etc. is tried.

[0003] The alloy which combined various elements according to the application is known by such hydrogen storing metal alloy, and a lanthanum-nickel system, a titanium-iron system, a zirconium-manganese system, a magnesium-nickel system, etc. are mentioned to it as a typical alloy. In these hydrogen storing metal alloys, the temperature dissociation pressure indicates one atmospheric pressure to be is 200 degrees C or more, and since occlusion of the hydrogen is carried out into an alloy at an elevated temperature, the occlusion and emission of hydrogen of a magnesium system alloy are attained using a hot heat source. Therefore, this magnesium system alloy is useful as the cold and the object for heating systems using for example, works

waste heat, or an object for hydrogen fueled cars using engine waste heat. The alloy which combined rare earth elements, such as others, magnesium and a lanthanum, and a cerium, as such a magnesium system hydrogen storing metal alloy is known. [alloy / of the conventional above / magnesium-nickel system]

[0004] on the other hand, the alloy of an yttrium and magnesium is indicated or reported by JP,57-45817,B, the collection 2nd separate volume of the 28th time autumn convention research presentation lecture summaries of the Society of Chemical Engineers, Japan, and the 345th page "the hydrogen permeation property of a Mg-Y alloy", for example — **** — it does not pass and the actual condition is hardly inquired. Moreover, it is almost unstated and is not known [in / approach / of this alloy / manufacture / the above-mentioned official report and the collection of summaries] about the solution process or the solidifying method for homogenization.

[0005] By the way, if an yttrium and magnesium compare those melting points, to the former being 1526 degrees C, the latter is 649 degrees C and has the big difference. Therefore, it is the metal which dissolves with a fusion furnace and is generally hard to alloy to a predetermined presentation. For example, if it is going to dissolve an yttrium and magnesium with a fusion furnace, it will follow on carrying out a temperature up, magnesium will dissolve first, and it will sediment to crucible, but before beginning to sublimate this magnesium to coincidence and forming a magnesium-yttrium alloy, the yttrium for a mind handbill and un-dissolving remains in the bottom of crucible, and magnesium invites the situation which is not desirable where the alloy which has a predetermined presentation cannot be formed. Although it is possible to blend too much the magnesium of the amount which is equivalent to a lost part beforehand in order to avoid such a situation that is not desirable, the amount which **** in this case is not fixed by dissolution conditions, as a result, dispersion arises in the hydrogen absorption property of the alloy obtained, or the problem of the engine performance fully not being demonstrated arises.

[0006]

[Problem(s) to be Solved by the Invention] Under such a situation, by the usual approach, this invention alloys the big magnesium and the big yttrium of a melting point difference with homogeneous difficult alloying to homogeneity so that a bias may not arise in a presentation, and it aims at offering the approach of manufacturing efficiently the magnesium-yttrium system hydrogen storing metal alloy which has the desired engine performance.

[0007]

[Means for Solving the Problem] When this invention persons did cooling coagulation

of that an alloy with a homogeneous presentation is efficiently obtained by using a specific solution process in case magnesium and an yttrium are alloyed, as a result of repeating research wholeheartedly, in order to attain said purpose, and the alloy molten metal formed further, they found out that the alloy which has the crystal structure with a homogeneously precise presentation was obtained by adopting a specific cooling rate. This invention is completed based on this knowledge.

[0008] Namely, this invention is in charge of manufacturing the hydrogen storing metal alloy which uses (1) magnesium and an yttrium as a principal component. (a) The first process which inserts an yttrium raw material in a fusion furnace, and is heated at the temperature below the melting point, (b) The second process which adds a magnesium raw material and carries out the heating dissolution so that it may become a predetermined presentation ratio in the heating raw material of the first process, And the manufacture approach of the magnesium-yttrium system hydrogen storing metal alloy characterized by performing the third process which carries out cooling coagulation of the magnesium-yttrium system alloy molten metal formed at the (c) second process one by one is offered. Moreover, this invention is in charge of manufacturing the hydrogen storing metal alloy which uses (2) magnesium and an yttrium as a principal component. Some of yttrium raw materials and magnesium raw materials are inserted in a fusion furnace. (a') In the heating raw material of the first process and the first (b') process heated at the temperature below the melting point of this yttrium raw material The second process which adds the remainder of a magnesium raw material and carries out the heating dissolution so that it may become a predetermined presentation ratio, And the manufacture approach of the magnesium-yttrium system hydrogen storing metal alloy characterized by performing the third process which carries out cooling coagulation of the magnesium-yttrium system alloy molten metal formed at the (c) second process one by one is also offered.

[0009] Furthermore, the desirable mode for carrying out this invention (3) In case a magnesium raw material is added and the heating dissolution is carried out in the second process, so that a rapid pressure buildup may not happen into a fusion furnace The above (1), the manufacture approach given in (2) of carrying out the heating dissolution, while controlling the climbing speed of furnace internal pressure, (4) The manufacture approach of the above-mentioned (3) publication which controls the pressure climbing speed in a fusion furnace to a part for 10 - 50Torr/, (5) The above which performs control of the pressure climbing speed in a fusion furnace by controlling the programming rate in a furnace (3), (4) In the manufacture approach of the above-mentioned (5) publication which controls the manufacture approach of a

publication, and the programming rate in (6) fusion furnaces to a part for 3 - 12-degree-C/, and the (7) third process the manufacture approach given [above-mentioned] in (1) - (6) of cooling with the cooling rate of 50-500 degrees C/second, and making a magnesium-yttrium system alloy molten metal solidifying -- it comes out.

[0010]

[Embodiment of the Invention] the alloy which the magnesium-yttrium system hydrogen storing metal alloy obtained by the approach of this invention uses magnesium and an yttrium as a principal component, and consists only of magnesium and an yttrium -- you may be -- these -- the suitable metal which can be alloyed further, for example, calcium, barium, strontium, aluminum, copper, and nickel -- it was further chosen from rare earth elements, such as a lanthanum and a cerium, etc. -- a kind may be made to contain at least In this invention, as a magnesium raw material used as one of the raw materials of the above-mentioned magnesium-yttrium system alloy, metal magnesium may be used and a Magnesium alloy, for example, a magnesium-calcium alloy etc., may be used depending on an alloy presentation. On the other hand, as an yttrium raw material used as another raw material, a metal yttrium may be used and an yttrium alloy, for example, an yttrium-rare-earth-elements alloy etc., may be used depending on an alloy presentation. Furthermore, the suitable metal or suitable alloy chosen from the above-mentioned metals with the magnesium raw material and the yttrium raw material can be used by the case.

[0011] Moreover, when alloying with a magnesium raw material and an yttrium raw material using other metals, In other metals, elements, such as the thing which has descriptions closer to magnesium than an yttrium, such as the melting point, for example, calcium, and aluminum It is desirable to adopt the same way of dealing with it as a magnesium raw material, and, as for elements, such as the thing which has descriptions closer to an yttrium than magnesium, such as the melting point, for example, a lanthanum, nickel, and copper, on the other hand, it is desirable to adopt the same way of dealing with it as an yttrium raw material.

[0012] The manufacture approach of the magnesium-yttrium system alloy of this invention consists of 3 of the first process shown below, the second process, and the third process processes. First, in the first process, all of yttrium raw materials or all of yttrium raw materials and the part of a magnesium raw material, and after inserting in the magnesium raw material of 30% or less of amount of loadings preferably and carrying out evacuation preferably, inert gas, such as argon gas, is introduced into the

crucible in fusion furnaces, such as a RF induction fusion furnace and a vacuum melting furnace, and the pressure in a furnace is maintained to extent preferably exceeding 200Torr(s) at it. Subsequently, in the temperature below the melting point of an yttrium raw material, it heats and the temperature up of the raw material inserted in in crucible is carried out until this raw material becomes red-hot. Under the present circumstances, the temperature of the above-mentioned raw material is more than the melting point of magnesium, and it is advantageous to heat so that it may become the range of 650-900 degrees C.

[0013] Next, in the second process, on the mixture of the yttrium raw material or yttrium raw material heated at the first above-mentioned process, and a magnesium raw material, it adds so that it may become a predetermined presentation ratio, and the temperature up of the magnesium raw material is carried out gradually. In this case, in the first process, since the raw material in crucible is heated more than the melting point of a magnesium raw material, the added magnesium raw material begins to dissolve gradually, and melting the part in contact with an yttrium raw material as an alloy in connection with it, the sedimentation dissolution is carried out at a crucible bottom, and it alloys to homogeneity.

[0014] In this second process, when a temperature up is performed rapidly, magnesium bumps, it scatters in a furnace or there is a possibility of causing the situations which are not desirable, such as ****(ing). In such a case, since the pressure in a furnace goes abruptly up, it is desirable to control a programming rate so that a rapid pressure buildup may not happen. It is advantageous to control a programming rate in 3-12-degree-C range for /, and to hold down a pressure climbing speed to the range for 10 - 50Torr/especially. A programming rate exceeds a part for 12-degree-C/, or if a pressure climbing speed exceeds a part for 50Torr(s)/, magnesium will bump, a **** loss becomes large, and the alloy of a homogeneity presentation is hard to be obtained. Moreover, it takes [alloying] time amount too much and is not desirable, when a programming rate is the following by 3-degree-C/or a pressure climbing speed is the following by 10Torr(s)/. In this second process, the range of the upper limit of whenever [furnace temperature] is usually 1050-1100 degrees C, and the upper limit of furnace internal pressure is usually the range of 300 - 400Torr. Since magnesium carries out the sedimentation dissolution at a crucible bottom, melting as an alloy the part which can lengthen the contact time of magnesium and an yttrium and contacts an yttrium raw material by carrying out the heating dissolution on such conditions, the uniform dissolution is attained.

[0015] Finally, in the third process, cooling coagulation of the magnesium-yttrium

system alloy molten metal formed at the second process of the above is carried out. It is desirable to perform this cooling coagulation on a water-cooled surface plate. Moreover, it is advantageous in this case to make it solidify with the cooling rate of 50–500 degrees C/second. In a second, the crystalline structure of an alloy does not do [this cooling rate] eburnation in less than 50 degrees C /, but there is a possibility that a hydrogen absorption property may not fully be demonstrated. Moreover, if a cooling rate exceeds a second in 500 degrees C /, turbulence and a good hydrogen absorption property may not be demonstrated for the crystal structure of an alloy. The range of 100–300 degrees C/second is suitable for the point of carrying out eburnation of the crystal structure of an alloy, and demonstrating a good hydrogen absorption property to especially a cooling rate. Thus, the obtained magnesium–yttrium system hydrogen storing metal alloy has a uniform presentation or a homogeneous presentation, and the precise crystal structure, and a desired hydrogen absorption property is demonstrated.

[0016]

[Example] Next, although an example explains this invention to a detail further, this invention is not limited at all by these examples.

Example 1Y5 Mg24 A total (99.9% of purity) of an yttrium (99.9% of purity) and 2000g of magnesium was used so that it might become an alloy presentation. First, after inserting the yttrium whole quantity in the crucible in a RF induction fusion furnace and exhausting the inside of a furnace to 0.1Torr(s), argon gas was introduced to 260Torr(s) and heating was started. When it heated for 40 minutes with about 20-degree-C programming rate for /and the temperature in a furnace amounted to 800 degrees C, the magnesium whole quantity was added to the yttrium in the crucible in a furnace. Subsequently, acting as a monitor so that the climbing speed of furnace internal pressure may become the range which is a part for 10 – 50Torr/, when it heated for 35 minutes and became 1070 degrees C with 8-degree-C programming rate for /, heating was stopped, and cooling coagulation of the alloy molten metal was carried out on conditions with a cooling rate of 150 degrees C [/second] on the water-cooled surface plate. Thus, the sampling was performed from the upper part and the lower part of the obtained alloy, ICP (Inductively Coupled Plazma) spectrographic analysis was performed, and the presentation was searched for. A result is shown in the 1st table.

[0017] Moreover, the scanning electron microscope (SEM) photograph Fig. of this alloy is shown in drawing 1 , and the qualitative point analysis chart by the EDS equipment of this alloy is shown in drawing 2 . The peak of magnesium and the peak of

an yttrium are accepted in this drawing 2 . Therefore, these drawings show that it is the precise magnesium-yttrium alloy of the crystal structure. Furthermore, the hydrogen absorption property of this alloy was searched for as follows.

[0018] argon gas after inserting this whole quantity in the crucible in a RF induction fusion furnace and exhausting the inside of a furnace to 0.1Torr(s) like example of comparison 1 example 1, using an yttrium and magnesium a total of 2000g — 260Torr(s) up to — it introduced and heating was started. It heats for 70 minutes with about 15-degree-C programming rate for /, and when molten metal temperature amounted to 1070 degrees C, cooling coagulation of the alloy molten metal was carried out on the water-cooled surface plate. Thus, the sampling was performed from the upper part and the lower part of the obtained alloy, ICP AEM was performed, and the presentation was searched for. A result is shown in the 1st table.

[0019] A total (99.9% of purity) of an yttrium (99.9% of purity), magnesium (99.9% of purity), and 2000g of copper was used so that it might become the alloy presentation of example 2Y5 Mg22.5 Cu1.5. First, after inserting the whole quantity of an yttrium and copper in the crucible in a RF induction fusion furnace and exhausting the inside of a furnace to 0.1Torr(s), argon gas was introduced to 260Torr(s) and heating was started. When it heated for 50 minutes with about 16-degree-C programming rate for /and the temperature in a furnace amounted to 800 degrees C, the magnesium whole quantity was added into the yttrium in the crucible in a furnace, and copper mixture. Subsequently, acting as a monitor so that the climbing speed of furnace internal pressure may become the range which is a part for 10 – 50Torr/, when it heated for 40 minutes and became 1070 degrees C with 7-degree-C programming rate for /, heating was stopped, and cooling coagulation of the alloy molten metal was carried out on conditions with a cooling rate of 150 degrees C [/second] on the water-cooled surface plate. Thus, the sampling was performed from the upper part and the lower part of the obtained alloy, ICP AEM was performed, and the presentation was searched for. A result is shown in the 1st table.

[0020] After inserting this whole quantity in the crucible in a RF induction fusion furnace and exhausting the inside of a furnace to 0.1Torr(s) like example of comparison 2 example 2, using an yttrium, magnesium, and copper a total of 2000g, argon gas was introduced to 260Torr(s) and heating was started. It heats for 85 minutes with about 13-degree-C programming rate for /, and when molten metal temperature amounted to 1070 degrees C, cooling coagulation of the alloy molten metal was carried out on the water-cooled surface plate. Thus, the sampling was performed from the upper part and the lower part of the obtained alloy, ICP AEM was

performed, and the presentation was searched for. A result is shown in the 1st table.

[0021]

[Table 1]

[0022] As shown in the 1st table, according to the approach of this invention, any element serves as a target system mostly, and does not almost have dispersion in a presentation in the upper part and the lower part of an alloy. On the other hand, by the approach of the example of a comparison, it separates greatly from a target system, there is dispersion in a presentation in the upper part and the lower part of an alloy, and the non-melt of an yttrium remained in the crucible bottom.

[0023]

[Effect of the Invention] According to the approach of this invention, magnesium can lose or the magnesium-yttrium system hydrogen storing metal alloy which un-dissolving [the amount of / of an yttrium] does not remain, and has a homogeneous presentation or a homogeneous presentation, and the precise crystal structure can be manufactured efficiently. The magnesium-yttrium system hydrogen storing metal alloy obtained by the approach of this invention is useful as an object for hydrogen fueled cars using the waste heat of the cold and the object for heating systems which has the desired hydrogen absorption property, for example, used works waste heat, or an engine etc.

TECHNICAL FIELD

[Field of the Invention] This invention relates to the approach of manufacturing efficiently the magnesium-yttrium system hydrogen storing metal alloy of a homogeneity presentation of the type used for hydrogen storage, heat pump, a cell, etc. in more detail by the available high temperature which has equilibrium pressure from ordinary pressure at 10atm(s), about the manufacture approach of a magnesium-yttrium system hydrogen storing metal alloy.

PRIOR ART

[Description of the Prior Art] In recent years, a hydrogen storing metal alloy attracts attention as one of the metal system new materials, and the application study is made positively. If it generates heat while it will absorb hydrogen and will serve as metal hydride, if this hydrogen storing metal alloy makes temperature low or a pressure is applied, and temperature is conversely made high or a pressure is lowered, it has the property to take heat at the same time it emits the hydrogen which was being absorbed again. Therefore, using such a property, a hydrogen storing metal alloy can be used for storage of hydrogen, storage of energy, or conversion of chemical energy and heat energy, for example, the use to hydrogen storage equipment, the heat pump of exhaust heat use, a chemical engine, the electrode material of a nickel hydride battery, etc. is tried.

[0003] The alloy which combined various elements according to the application is known by such hydrogen storing metal alloy, and a lanthanum-nickel system, a titanium-iron system, a zirconium-manganese system, a magnesium-nickel system, etc. are mentioned to it as a typical alloy. In these hydrogen storing metal alloys, the temperature dissociation pressure indicates one atmospheric pressure to be is 200 degrees C or more, and since occlusion of the hydrogen is carried out into an alloy at an elevated temperature, the occlusion and emission of hydrogen of a magnesium system alloy are attained using a hot heat source. Therefore, this magnesium system alloy is useful as the cold and the object for heating systems using for example, works waste heat, or an object for hydrogen fueled cars using engine waste heat. The alloy which combined rare earth elements, such as others, magnesium and a lanthanum, and a cerium, as such a magnesium system hydrogen storing metal alloy is known. [alloy / of the conventional above / magnesium-nickel system]

[0004] on the other hand, the alloy of an yttrium and magnesium is indicated or reported by JP,57-45817,B, the collection 2nd separate volume of the 28th time autumn convention research presentation lecture summaries of the Society of

Chemical Engineers, Japan, and the 345th page "the hydrogen permeation property of a Mg-Y alloy", for example — **** — it does not pass and the actual condition is hardly inquired. Moreover, it is almost unstated and is not known [in / approach / of this alloy / manufacture / the above-mentioned official report and the collection of summaries] about the solution process or the solidifying method for homogenization. [0005] By the way, if an yttrium and magnesium compare those melting points, to the former being 1526 degrees C, the latter is 649 degrees C and has the big difference. Therefore, it is the metal which dissolves with a fusion furnace and is generally hard to alloy to a predetermined presentation. For example, if it is going to dissolve an yttrium and magnesium with a fusion furnace, it will follow on carrying out a temperature up, magnesium will dissolve first, and it will sediment to crucible, but before beginning to sublimate this magnesium to coincidence and forming a magnesium-yttrium alloy, the yttrium for a mind handbill and un-dissolving remains in the bottom of crucible, and magnesium invites the situation which is not desirable where the alloy which has a predetermined presentation cannot be formed. Although it is possible to blend too much the magnesium of the amount which is equivalent to a lost part beforehand in order to avoid such a situation that is not desirable, the amount which **** in this case is not fixed by dissolution conditions, as a result, dispersion arises in the hydrogen absorption property of the alloy obtained, or the problem of the engine performance fully not being demonstrated arises.

EFFECT OF THE INVENTION

[Effect of the Invention] According to the approach of this invention, magnesium can lose or the magnesium-yttrium system hydrogen storing metal alloy which un-dissolving [the amount of / of an yttrium] does not remain, and has a homogeneous presentation or a homogeneous presentation, and the precise crystal structure can be manufactured efficiently. The magnesium-yttrium system hydrogen storing metal alloy obtained by the approach of this invention is useful as an object for hydrogen fueled cars using the waste heat of the cold and the object for heating systems which has the desired hydrogen absorption property, for example, used works waste heat, or an engine etc.

TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] Under such a situation, by the usual

approach, this invention alloys the big magnesium and the big yttrium of a melting point difference with homogeneous difficult alloying to homogeneity so that a bias may not arise in a presentation, and it aims at offering the approach of manufacturing efficiently the magnesium-yttrium system hydrogen storing metal alloy which has the desired engine performance.

MEANS

[Means for Solving the Problem] When this invention persons did cooling coagulation of that an alloy with a homogeneous presentation is efficiently obtained by using a specific solution process in case magnesium and an yttrium are alloyed, as a result of repeating research wholeheartedly, in order to attain said purpose, and the alloy molten metal formed further, they found out that the alloy which has the crystal structure with a homogeneously precise presentation was obtained by adopting a specific cooling rate. This invention is completed based on this knowledge.

[0008] Namely, this invention is in charge of manufacturing the hydrogen storing metal alloy which uses (1) magnesium and an yttrium as a principal component. (a) The first process which inserts an yttrium raw material in a fusion furnace, and is heated at the temperature below the melting point, (b) The second process which adds a magnesium raw material and carries out the heating dissolution so that it may become a predetermined presentation ratio in the heating raw material of the first process, And the manufacture approach of the magnesium-yttrium system hydrogen storing metal alloy characterized by performing the third process which carries out cooling coagulation of the magnesium-yttrium system alloy molten metal formed at the (c) second process one by one is offered. Moreover, this invention is in charge of manufacturing the hydrogen storing metal alloy which uses (2) magnesium and an yttrium as a principal component. Some of yttrium raw materials and magnesium raw materials are inserted in a fusion furnace. (a') In the heating raw material of the first process and the first (b') process heated at the temperature below the melting point of this yttrium raw material The second process which adds the remainder of a magnesium raw material and carries out the heating dissolution so that it may become a predetermined presentation ratio, And the manufacture approach of the magnesium-yttrium system hydrogen storing metal alloy characterized by performing the third process which carries out cooling coagulation of the magnesium-yttrium system alloy molten metal formed at the (c) second process one by one is also offered.

[0009] Furthermore, the desirable mode for carrying out this invention (3) In case a

magnesium raw material is added and the heating dissolution is carried out in the second process, so that a rapid pressure buildup may not happen into a fusion furnace. The above (1), the manufacture approach given in (2) of carrying out the heating dissolution, while controlling the climbing speed of furnace internal pressure, (4) The manufacture approach of the above-mentioned (3) publication which controls the pressure climbing speed in a fusion furnace to a part for 10 – 50Torr/, (5) The above which performs control of the pressure climbing speed in a fusion furnace by controlling the programming rate in a furnace (3), (4) In the manufacture approach of the above-mentioned (5) publication which controls the manufacture approach of a publication, and the programming rate in (6) fusion furnaces to a part for 3 – 12-degree-C/, and the (7) third process the manufacture approach given [above-mentioned] in (1) – (6) of cooling with the cooling rate of 50–500 degrees C/second, and making a magnesium-yttrium system alloy molten metal solidifying -- it comes out.

[0010]

[Embodiment of the Invention] the alloy which the magnesium-yttrium system hydrogen storing metal alloy obtained by the approach of this invention uses magnesium and an yttrium as a principal component, and consists only of magnesium and an yttrium -- you may be -- these -- the suitable metal which can be alloyed further, for example, calcium, barium, strontium, aluminum, copper, and nickel -- it was further chosen from rare earth elements, such as a lanthanum and a cerium, etc. -- a kind may be made to contain at least In this invention, as a magnesium raw material used as one of the raw materials of the above-mentioned magnesium-yttrium system alloy, metal magnesium may be used and a Magnesium alloy, for example, a magnesium-calcium alloy etc., may be used depending on an alloy presentation. On the other hand, as an yttrium raw material used as another raw material, a metal yttrium may be used and an yttrium alloy, for example, an yttrium-rare-earth-elements alloy etc., may be used depending on an alloy presentation. Furthermore, the suitable metal or suitable alloy chosen from the above-mentioned metals with the magnesium raw material and the yttrium raw material can be used by the case.

[0011] Moreover, when alloying with a magnesium raw material and an yttrium raw material using other metals, In other metals, elements, such as the thing which has descriptions closer to magnesium than an yttrium, such as the melting point, for example, calcium, and aluminum It is desirable to adopt the same way of dealing with it as a magnesium raw material, and, as for elements, such as the thing which has

descriptions closer to an yttrium than magnesium, such as the melting point, for example, a lanthanum, nickel, and copper, on the other hand, it is desirable to adopt the same way of dealing with it as an yttrium raw material.

[0012] The manufacture approach of the magnesium-yttrium system alloy of this invention consists of 3 of the first process shown below, the second process, and the third process processes. First, in the first process, all of yttrium raw materials or all of yttrium raw materials and the part of a magnesium raw material, and after inserting in the magnesium raw material of 30% or less of amount of loadings preferably and carrying out evacuation preferably, inert gas, such as argon gas, is introduced into the crucible in fusion furnaces, such as a RF induction fusion furnace and a vacuum melting furnace, and the pressure in a furnace is maintained to extent preferably exceeding 200Torr(s) at it. Subsequently, in the temperature below the melting point of an yttrium raw material, it heats and the temperature up of the raw material inserted in in crucible is carried out until this raw material becomes red-hot. Under the present circumstances, the temperature of the above-mentioned raw material is more than the melting point of magnesium, and it is advantageous to heat so that it may become the range of 650-900 degrees C.

[0013] Next, in the second process, on the mixture of the yttrium raw material or yttrium raw material heated at the first above-mentioned process, and a magnesium raw material, it adds so that it may become a predetermined presentation ratio, and the temperature up of the magnesium raw material is carried out gradually. In this case, in the first process, since the raw material in crucible is heated more than the melting point of a magnesium raw material, the added magnesium raw material begins to dissolve gradually, and melting the part in contact with an yttrium raw material as an alloy in connection with it, the sedimentation dissolution is carried out at a crucible bottom, and it alloys to homogeneity.

[0014] In this second process, when a temperature up is performed rapidly, magnesium bumps, it scatters in a furnace or there is a possibility of causing the situations which are not desirable, such as ****(ing). In such a case, since the pressure in a furnace goes abruptly up, it is desirable to control a programming rate so that a rapid pressure buildup may not happen. It is advantageous to control a programming rate in 3-12-degree-C range for /, and to hold down a pressure climbing speed to the range for 10 - 50Torr/especially. A programming rate exceeds a part for 12-degree-C/, or if a pressure climbing speed exceeds a part for 50Torr(s)/, magnesium will bump, a **** loss becomes large, and the alloy of a homogeneity presentation is hard to be obtained. Moreover, it takes [alloying] time amount too

much and is not desirable, when a programming rate is the following by 3-degree-C/or a pressure climbing speed is the following by 10Torr(s)/. In this second process, the range of the upper limit of whenever [furnace temperature] is usually 1050-1100 degrees C, and the upper limit of furnace internal pressure is usually the range of 300 - 400Torr. Since magnesium carries out the sedimentation dissolution at a crucible bottom, melting as an alloy the part which can lengthen the contact time of magnesium and an yttrium and contacts an yttrium raw material by carrying out the heating dissolution on such conditions, the uniform dissolution is attained.

[0015] Finally, in the third process, cooling coagulation of the magnesium-yttrium system alloy molten metal formed at the second process of the above is carried out. It is desirable to perform this cooling coagulation on a water-cooled surface plate. Moreover, it is advantageous in this case to make it solidify with the cooling rate of 50-500 degrees C/second. In a second, the crystalline structure of an alloy does not do [this cooling rate] eburation in less than 50 degrees C /, but there is a possibility that a hydrogen absorption property may not fully be demonstrated. Moreover, if a cooling rate exceeds a second in 500 degrees C /, turbulence and a good hydrogen absorption property may not be demonstrated for the crystal structure of an alloy. The range of 100-300 degrees C/second is suitable for the point of carrying out eburation of the crystal structure of an alloy, and demonstrating a good hydrogen absorption property to especially a cooling rate. Thus, the obtained magnesium-yttrium system hydrogen storing metal alloy has a uniform presentation or a homogeneous presentation, and the precise crystal structure, and a desired hydrogen absorption property is demonstrated.

EXAMPLE

[Example] Next, although an example explains this invention to a detail further, this invention is not limited at all by these examples.

Example 1Y5 Mg24 A total (99.9% of purity) of an yttrium (99.9% of purity) and 2000g of magnesium was used so that it might become an alloy presentation. First, after inserting the yttrium whole quantity in the crucible in a RF induction fusion furnace and exhausting the inside of a furnace to 0.1Torr(s), argon gas was introduced to 260Torr(s) and heating was started. When it heated for 40 minutes with about 20-degree-C programming rate for /and the temperature in a furnace amounted to 800 degrees C, the magnesium whole quantity was added to the yttrium in the crucible in a furnace. Subsequently, acting as a monitor so that the climbing speed of furnace

internal pressure may become the range which is a part for 10 – 50Torr/, when it heated for 35 minutes and became 1070 degrees C with 8-degree-C programming rate for /, heating was stopped, and cooling coagulation of the alloy molten metal was carried out on conditions with a cooling rate of 150 degrees C [/second] on the water-cooled surface plate. Thus, the sampling was performed from the upper part and the lower part of the obtained alloy, ICP (Inductively Coupled Plasma) spectrographic analysis was performed, and the presentation was searched for. A result is shown in the 1st table.

[0017] Moreover, the scanning electron microscope (SEM) photograph Fig. of this alloy is shown in drawing 1 , and the qualitative point analysis chart by the EDS equipment of this alloy is shown in drawing 2 . The peak of magnesium and the peak of an yttrium are accepted in this drawing 2 . Therefore, these drawings show that it is the precise magnesium-yttrium alloy of the crystal structure. Furthermore, the hydrogen absorption property of this alloy was searched for as follows.

[0018] argon gas after inserting this whole quantity in the crucible in a RF induction fusion furnace and exhausting the inside of a furnace to 0.1Torr(s) like example of comparison 1 example 1, using an yttrium and magnesium a total of 2000g — 260Torr(s) up to — it introduced and heating was started. It heats for 70 minutes with about 15-degree-C programming rate for /, and when molten metal temperature amounted to 1070 degrees C, cooling coagulation of the alloy molten metal was carried out on the water-cooled surface plate. Thus, the sampling was performed from the upper part and the lower part of the obtained alloy, ICP AEM was performed, and the presentation was searched for. A result is shown in the 1st table.

[0019] A total (99.9% of purity) of an yttrium (99.9% of purity), magnesium (99.9% of purity), and 2000g of copper was used so that it might become the alloy presentation of example 2Y5 Mg22.5 Cu1.5. First, after inserting the whole quantity of an yttrium and copper in the crucible in a RF induction fusion furnace and exhausting the inside of a furnace to 0.1Torr(s), argon gas was introduced to 260Torr(s) and heating was started. When it heated for 50 minutes with about 16-degree-C programming rate for /and the temperature in a furnace amounted to 800 degrees C, the magnesium whole quantity was added into the yttrium in the crucible in a furnace, and copper mixture. Subsequently, acting as a monitor so that the climbing speed of furnace internal pressure may become the range which is a part for 10 – 50Torr/, when it heated for 40 minutes and became 1070 degrees C with 7-degree-C programming rate for /, heating was stopped, and cooling coagulation of the alloy molten metal was carried out on conditions with a cooling rate of 150 degrees C [/second] on the water-cooled

surface plate. Thus, the sampling was performed from the upper part and the lower part of the obtained alloy, ICP AEM was performed, and the presentation was searched for. A result is shown in the 1st table.

[0020] After inserting this whole quantity in the crucible in a RF induction fusion furnace and exhausting the inside of a furnace to 0.1Torr(s) like example of comparison 2 example 2, using an yttrium, magnesium, and copper a total of 2000g, argon gas was introduced to 260Torr(s) and heating was started. It heats for 85 minutes with about 13-degree-C programming rate for /, and when molten metal temperature amounted to 1070 degrees C, cooling coagulation of the alloy molten metal was carried out on the water-cooled surface plate. Thus, the sampling was performed from the upper part and the lower part of the obtained alloy, ICP AEM was performed, and the presentation was searched for. A result is shown in the 1st table.

[0021]

[Table 1]

No translation

[0022] As shown in the 1st table, according to the approach of this invention, any element serves as a target system mostly, and does not almost have dispersion in a presentation in the upper part and the lower part of an alloy. On the other hand, by the approach of the example of a comparison, it separates greatly from a target system, there is dispersion in a presentation in the upper part and the lower part of an alloy, and the non-melt of an yttrium remained in the crucible bottom.

DESCRIPTION OF DRAWINGS

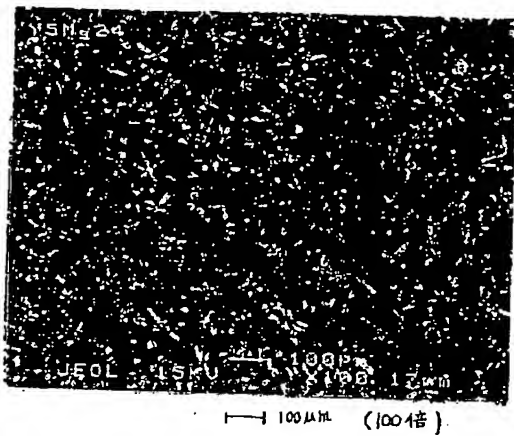
[Brief Description of the Drawings]

[Drawing 1] It is the scanning electron microscope (SEM) photograph Fig. of the magnesium-yttrium alloy obtained in the example 1.

[Drawing 2] It is a qualitative point analysis chart by the EDS equipment of the magnesium-yttrium alloy obtained in the example 1.

DRAWINGS

[Drawing 1]



[Drawing 2]

